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MANUAL

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Pharmaceutical Laboratory

SEBUFFALD:

COLLEGE OF PHARMACY.

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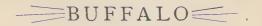
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MANUAL

FOR THE

Pharmaceutical Laboratory

OF THE



COLLEGE OF PHARMACY.



WILLIS G. GREGORY, M. D., Ph. G.,



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NOTE TO STUDENTS.

The lessons in this Manual will be found divided into two parts: "Galenical Preparations" and "Chemical Preparations." Subordinate groups will also be noticed, such as Ointments, Syrups, Abstracts, and preparations made by drying, calcination, scaling, etc This arrangement is chosen to call the attention of students to the classification of pharmaceutical operations.

It is desirable in the laboratory to have the lessons so follow each other that a variety of operations can be carried on at the same time, employing the different pieces of apparatus at command. If this is not done, a loss of time necessarily occurs in making successively a number of preparations requiring the same set of utensils.

It is, therefore, not expected that the numerical order of the lessons will be observed in laboratory work. It is suggested that they be studied according to the following arrangement:

$$\begin{array}{c} 1-2-3-4-31-32-5-6-33-34-35-7-8-9-37\\ -36-10-11-38-12-13-39-14-15-16-40-17-18\\ -19-20-41-42-44-45-43-21-22-23-24-46-47\\ -25-26-27-48-49-50-51-52-53-28-29-54-55\\ -56-57-58-30-59-60. \end{array}$$

PART I.

GALENICAL PREPARATIONS.

MEDICATED WATERS.

I.—AQUA CAMPHORÆ, U. S. P.:

| Campho | r | | | | | | | | | | 2 Gm. |
|-------------|---|---|----|----|--|-----|-----|----|----|--|---------|
| Alcohol | | | | | | | | | ų. | | 4 Gm. |
| $Cotton\ .$ | | | | | | ٠ | | | | | 4 Gm. |
| Distilled | 7 | V | at | er | | . Ç | 2.9 | 5. | | | 250 Gm. |

Dissolve camphor in alcohol. Add this solution to cotton in small portions, distributing well by picking cotton thoroughly after each addition. After alcohol has nearly all evaporated, pack cotton firmly in percolator, and gradually pour on water until 250 Gm. (250 C.c.) of percolate are obtained. If first portion of percolate is not clear, return it to percolator, and repeat until product is perfectly clear.

2.—AQUA CINNAMOMI:

| Oil of Cinnamon | | | | | 0.5 | l.c. |
|-----------------|-----|-----|--|--|-----|------|
| Aqua Fervens | . Q | .S. | | | 250 | i.c. |

Add the oil to the water, and shake frequently until thoroughly cold. Filter, returning the first portion of the filtrate to the filter if not clear, and adding sufficient cold water to make the product measure 250 C.c.



| 3.—AQUA GAULTHERIÆ (PROCESS OF 1870): |
|--|
| Oil of Wintergreen |
| ually, rubbing well for some time. Add portion of water (50 C.c.), stirring well during addition. Transfer to filter, and pass enough water through to make 250 C.c. If first filtrate is not clear, return to filter. |
| OINTMENTS. |
| 4.—Adeps Benzoinatus, U. S. P.: |
| Benzoin, in coarse powder 2 Gm. Lard |
| 6.—Unguentum Acidi Carbolici, U. S. P.: |
| Carbolic Acid 5 Gm. Ointment (use previous product— No. 5) |
| 7.—Unguentum Zinci Oxidi, U. S. P.: Oxide of Zinc (use No. 35) 10 Gm. Benzoinated Lard (use No. 4) 40 Gm. Rub zinc in warm mortar with 10 Gm. of lard, previously |
| |



melted, until perfectly smooth, then add remainder of lard, and mix with pestle until cold.

8.—Unguentum Iodi, U. S. P.:

| Iodine | | | | | | | | | | | | 2 | Gm. |
|--------|-----|----|-----|-----|----|----|----|----|----|----|--|------|-----|
| Iodide | of | Po | ota | ssi | ur | n | | | | | | 0.5 | Gm. |
| Water. | | | | | | | | | | | | 1 | Gm. |
| Benzoi | nat | ed | L | ar | d | (u | se | No |). | 4) | | 46.5 | Gm. |

Rub the iodine and iodide first with water, and then with the benzoinated lard, *gradually* added, until thoroughly mixed, avoiding use of iron spatula.

9.—Unguentum Hydrargyri Nitratis, U. S. P.:

| Mercury | | | | ٠ | | | 4 | | 3.5 | Gm. |
|-------------|---|-----|-----|---|----|--|---|--|-----|-----|
| Nitric Acid | | | | | ٠. | | | | 8.5 | Gm. |
| Lard Oil (4 | 2 | C.c | 2.) | | | | | | 38 | Gm. |

Heat the oil in capsule over water bath to 70° C. (158° F.) Then add, without stirring, 3.5 Gm. nitric acid. Continue heat as long as effervescence occurs, and allow mixture to cool. Dissolve the mercury in the balance of nitric acid in small capsule, with sufficient heat to prevent crystallization, and add to the mixture before cold. Mix thoroughly, avoiding iron spatula.

CERATES.

10.—CERATUM, U. S. P.:

| White | Wax. | | | | | | 12 Gm. |
|-------|------|--|--|--|--|--|--------|
| Lard | | | | | | | 28 Gm |

Melt the ingredients together and stir until cold. (Preserve for use.)



II.—CERATUM CAMPHORÆ, U. S. P.:

| Camphor Liniment | | | | 15 | Gm. |
|------------------|--|--|--|------|-----|
| Olive Oil | | | | 6 | Gm. |
| (Cerate No. 10) | | | | 42.5 | Gm. |

Mix the liniment and oil. Melt the cerate, and when cooling add the mixture, stirring only enough to thoroughly incorporate.

TINCTURES.

12.—TINCTURA NUCIS VOMICÆ, U. S. P.:

Mix alcohol and water in proportion of eight parts alcohol (98 C.c.) and one part water (10 C.c.). Moisten powder with 25 C.c. of this menstruum, mix well, breaking all lumps with spatula or fingers, place in percolator and allow to stand one hour. Then pack firmly, arrange rubber tube, pour on just enough menstruum to keep surface of drug covered, and macerate until next lesson. Then gradually pour menstruum upon it until 250 C.c. of percolate are obtained, not allowing flow of percolate to exceed twenty drops a minute. Mix the percolate thoroughly, and evaporate in weighed capsule on water bath, 50 Gm. (60 C.c.) of it to dryness, taking care not to burn the solid extract. When completely dry, weigh capsule with extract, and by subtracting weight of capsule, find amount of extract. This should be two per cent. (I Gm.) of the quantity evaporated. From extract obtained, calculate the amount in whole percolate. Dissolve dry extract in enough menstruum so that when added to remainder of percolate the finished tincture will contain two per cent. of dry extract. Mix and filter. State weight of extract obtained



and volume of finished tincture in laboratory record. (Preserve for use.)

Note.—This assay process can be applied to the tincture of nux vomica in your stores, and will show the value of the fluid extract or percolation by which it is made, provided the menstruum in each case is the official one.

FLUID EXTRACTS.

13.—Extractum Zingiberis Fluidum, U. S. P.:

Ginger (No. 40 powder) 50 Gm. Alcohol Q.S.

Prepare for percolation as previously instructed. Macerate until next lesson. Percolate not faster than fifteen drops per minute, until drug is exhausted. (How know this?) Reserve first 45 C.c. of percolate, and evaporate remainder to soft extract; dissolve this in reserved portion, and add alcohol to make finished product measure 50 C.c. (Preserve for use.)

SYRUPS.

14.—Syrupus Tolutanus, U. S. P.:

| Balsam Tolu | | | | | 4 Gm. |
|-----------------|--|--|--|--|--------|
| Sugar | | | | | 65 Gm. |
| Distilled Water | | | | | |

Mix sugar with 35 C.c. water, add tolu and digest in covered vessel at temperature not to exceed 82° C. (180° F.) for two hours. Let cool, and strain through wet muslin, adding, through strainer, enough water to make the syrup weigh 100 Gm. (80 C.c.) Mix thoroughly.

15.—Syrupus Tolutanus (1870):

| Balsam T | olu | | | | | | | | | 4 Gm. |
|-----------|------|--------------|----|----|------|----|--|--|--|--------|
| Alcohol . | | | | | | | | | | 10 Gm. |
| Carbonate | e of | \mathbf{M} | ag | ne | esia | 1. | | | | 3 Gm. |
| Sugar | | | , | | | | | | | 65 Gm. |
| Water. | | | | | | | | | | 70 Gm. |



Place alcohol and tolu in small flask, warm slightly, and dissolve by agitation. Rub this tincture first with magnesia, and 10 Gm. sugar, and then with the water gradually added, and filter. To the filtrate add the remainder of the sugar, dissolve with gentle heat and strain while hot. (Compare with official syrup.)

16.—Syrupus Rhei, U. S. P.:

| Rhubarb, sliced | | 18 Gm. |
|------------------------|------|---------|
| Cinnamon, bruised | | |
| Carbonate of Potassium | | 1.2 Gm. |
| Sugar | | 120 Gm. |
| Water | O.S. | 200 Gm. |

Wash rhubarb free from dust, then mix it with cinnamon, carbonate of potassium and 84 C.c. of water. Macerate mixture in closed vessel twelve hours, then strain and filter, adding through the dregs, if necessary, enough water to make filtered liquid weigh 80 Gm. (80 C.c.) Lastly add the sugar, dissolve by agitation without heat, and strain.

17.—SYRUPUS PRUNI VIRGINIANÆ, U. S. P.:

| Wild Cherry (No. | 20 powe | der) | 24 Gm. |
|------------------|---------|------|---------|
| Sugar | | | |
| Glycerine | | | 10 Gm. |
| Water | | O.S. | 200 Gm. |

Moisten the wild cherry with water, macerate in percolator twenty-four hours, then percolate with water until 70 C.c. of percolate are obtained. In this liquid dissolve the sugar by agitation without heat, add the glycerine, mix and strain.

18.—Syrupus Zingiberis, U. S. P.:

| Fluid Ex | tra | ct | of | G | in | gei | (| No | . : | 13) | | 4 | Gm. |
|----------|-----|----|----|---|----|-----|---|----|-----|-----|-----|-----|-----|
| Sugar | | | | | | | | | | | | 130 | Gm. |
| Water. | | | | | | | | | | . (| o.S | 200 | Gm. |

Rub the fluid extract with 50 Gm. of sugar, and, when



well mixed, warm very gently (60° C.) (140 C.c.) to evaporate alcohol. Dissolve this sugar by agitation in 70 C.c. water, filter, add through filter, water Q.S. to make the whole weigh 120 Gm. (100 C.c.), add remainder of sugar, dissolve by agitation without heat, and strain.

19.—Syrupus (By Percolation):

| Sugar. | | | | | | | | 200 | Gm. |
|--------|--|--|--|---|--|--|--|-----|-----|
| Water | | | | , | | | | IIO | Gm. |
| Cotton | | | | | | | | | Gm. |

Pack cotton moderately with handle of packer in percolator without cork or tube. Add sugar in portions, shaking down after each addition by tapping percolator on counter. Pour in the water and wait. If flow exceeds ten drops a minute, press cotton tighter in percolator with glass rod, and return first product. Continue percolation until all water has passed through. (Preserve for use.)

20.—Soluble Essence of Ginger:

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Fluid Extract of Ginger (No. 13) . . . 33 C.c. Powdered Pumice-stone . . . . . . . 15 Gm. Water . . . . . . . . Q.S. 100 Gm.
```

Shake fluid extract and pumice together, adding 65 C.c. water in about five portions, shaking well after each addition. Let stand twenty-four hours, then shake and filter, adding enough water through filter to make filtrate weigh 100 Gm. (100 C.c.) (Preserve for use.)

21.—Syrupus Zingiberis:

Estimate the amount of essence (No. 20) and the amount of syrup (No. 19) necessary to make 100 C.c. finished syrup of official strength. (See U. S. P. Syrupus Zingiberis, No. 18.)



ABSTRACTS.

| 22.—Abstractum Nucis Vomicæ, U. S. P.: | |
|--|---------|
| Nux Vomica (No. 60 powder) | 30 Gm |
| Sugar of Milk | ız Gm |
| Tracer | 15 0111 |

To save time, proceed as follows:

Evaporate 150 C. of tincture No. 12 to 25 C.c., and add 8 Gm. sugar of milk. Set aside in warm place, not above 50° C. (122° F.), covered with muslin, until the mixture is dry; add enough sugar of milk (4 Gm.) to make mixture weigh 15 Gm., powder thoroughly and preserve well-corked. (Preserve sample.)

23.—Abstractum Jalapæ, U. S. P.:

Moisten the jalap with 12 C.c. alcohol, macerate forty-eight hours, and percolate as previously instructed, not allowing flow to exceed fifteen drops per minute, until exhausted. (See U. S. P.) Of tincture thus obtained, take two-fifths and evaporate on water bath to 20 C.c. Add 5 Gm. sugar of milk, cover with muslin and set aside, in warm place, not above 50° C. (122° F.), until mixture is dry. Then add enough sugar of milk (4 Gm.) to make 10 Gm.; powder thoroughly and keep in well-corked bottle. (Preserve sample.)

RESINS.

| 24.—KESINA JALA | APÆ, | U. | 5. | P.: | | | | |
|-----------------|------|----|----|-----|--|--|-----|---|
| Jalap | | | | | | | | |
| Alcohol | | | | | | | | |
| Water | | | | | | | iΟ. | S |



Evaporate balance (three-fifths) of jalap percolate(No. 23) to 25 C.c. Add this gradually to 400 C.c. of cold water, stirring constantly. When the precipitate has formed, decant the clear liquid and wash the precipitate twice by decantation. Place it on strainer, express liquid and dry with a gentle heat. (Preserve sample.)

PILLS.

25.—PILULÆ RHEI, U. S. P.:

Rhubarb, in fine powder gr. 36
Soap, in fine powder gr. 12
To make twelve pills.

Beat them together with water so as to form a mass, and divide it into twelve pills.

26.—PILULÆ FERRI COMPOSITÆ, U. S. P.:

Myrrh, in fine powder gr. 18
Carbonate of Sodium gr. 9
Sulphate of Iron gr. 9
Syrup (No. 19) Q. S.

To make twelve pills. (Preserve sample.)

Rub the myrrh first with the carbonate, and afterward with the sulphate, until they are thoroughly mixed; then beat with syrup so as to form a mass, and divide it into twelve pills. (Preserve sample.)

EXTRACTS.

No. 27.—Extractum Gentianæ, U. S. P.:

Gentian (No. 20 powder) 40 Gm. Water. Q. S.

Prepare for percolation and macerate twenty-four hours. Let percolation proceed, not faster than thirty drops per minute, until exhaustion. Reduce percolate to three-quarters its weight by boiling, strain and then evaporate on water bath to pilular consistence.



STUDY IN PERCOLATION.

28.—RATE OF EXHAUSTION:

Taraxacum (No. 30 powder) 50 Gm. Diluted Alcohol O. S.

Prepare for percolation as previously directed, and macerate forty-eight hours. Then proceed with percolation, not allowing flow to exceed twenty drops per minute; collect percolate in fractions of 50 C.c. until five such portions are obtained, each 50 C.c. Number these portions in order, as obtained, 1, 2, 3, 4 and 5. Of portion No. 1 evaporate on water bath, in small weighed capsule, 25 C.c. to dry extract, and record weight. Treat each portion in same way. By comparing the amounts of dry extractive found in the successive portions with the total amount (the sum of the five weights), the rate of exhaustion or the proportion of dry extract in each 50 C.c. of percolate can be learned. Record the weights obtained.

29.-VALUE OF MACERATION:

Aconite Root in No. 60 powder . . 100 Gm. Alcohol Q. S.

Divide the aconite into two equal portions, marking same A and B. Prepare A for percolation and macerate forty-eight hours. Then proceed with percolation, not allowing flow to exceed thirty drops per minute, collect percolate in fractions of 50 C.c. until five such portions are obtained, each 50 C.c. Number these portions in order as obtained, A1, A2, A3, A4 and A5. Of portion A1, evaporate on water bath in small weighed capsule 25 C.c. to dry extract, and record weight. Treat each portion in same way. Now prepare B for percolation, and percolate at once without maceration, completing the operation in one session. Allow flow of



percolate to proceed at the same rate and collect in the same manner as with A, and mark in order as obtained, B₁, B₂, B₃, B₄ and B₅. Evaporate in same manner, and record dry extractive. By comparing the amounts of extractive matter in A₁ and B₁, and so on, the value of maceration with this drug can be learned. The rate of exhaustion by each method of percolation can be ascertained by comparing the amount of extractive in each portion of each series with the whole amount (the sum of the five weights) in the same series. Record all weights obtained.

EMULSIONS.

30.—Emulsio Olei Gossypii Seminis:

| Powdered Acacia . | | | | 15 Gm. |
|-------------------|--|--|--|---------|
| Powered Sugar | | | | 20 Gm. |
| Cotton Seed Oil. | | | | 50 C.c. |
| Water | | | | |

Place acacia and sugar in mortar and mix thoroughly. Gradually add 25 C.c. of distilled water, and triturate until smooth mucilage results. Then add the oil slowly and with constant rubbing, until thoroughly emulsified. Finally incorporate enough water (15 C.c.) to make product measure 100 C.c. Present for credit at next lesson without shaking.



PART II.

CHEMICAL PREPARATIONS.

DRY PROCESSES.

TRITURATION.

. 19 Gm.

| Iodine |
|---|
| Triturate iodine very thoroughly with little distilled water (3 C.c.); add starch gradually, and continue triturating until |
| the compound assumes a uniform blue color, approaching |
| black. Dry at ordinary temperature, and rub to a fine pow- |

32.—Hydrargyri Iodidi Viride, U. S. P.:

31.—AMYLUM IODATUM, U. S. P. :

der. (Preserve sample.)

| Mercury. | | | | | | | 8 Gm. |
|----------|--|--|--|--|--|--|-------|
| Iodine . | | | | | | | 5 Gm. |
| Alcohol | | | | | | | 0 9 |

Pour about 3 C.c. alcohol into a mortar containing the mercury, add iodine in several successive portions, and triturate the mixture, adding alcohol from time to time (2 C.c) to keep the mass moist, taking care that it be not exposed to



hight. Continue trituration until all globules of mercury disappear, and the mixture is nearly dry, and is greenish-yellow in color. Then add alcohol to reduce the whole to a thin paste, pour this into a bottle, let stand for several days in dark place, then wash the iodide twice with about 50 C.c. warm alcohol each time, and decant the washings. Transfer iodide to filter, and wash with warm alcohol (50 C.c.) Dry the product in a dark place, between filter paper, at ordinary temperature. Keep in bottles protected from light. (Preserve sample.)

DRYING.

33.—Alumen Exsiccatum, U. S. P.:

Alum 50 Gm.

Heat alum *gently* in porcelain capsule on sand bath until it dissolves, then gradually raise temperature until the mass becomes white and porous. When cold, powder. (Preserve sample.)

34.—FERRI SULPHAS EXSICCATUS, U. S. P.:

Sulphate of Iron 50 Gm.

Heat iron moderately in empty sand bath, occasionally stirring, until it has effloresced, then increase the heat, and keep hot until vapor ceases to be evolved. When cool, powder. (Preserve sample.)

CALCINATION.

35—ZINCI OXIDUM:

Carbonate of Zinc 16 Gm.

Heat dry, strongly, in clean, empty sand bath until water and carbonic acid are wholly expelled, and no effervescence is produced when a small portion is dropped into dilute sulphuric acid. (Preserve for use.)



36.—FERRI OXIDUM:

Oxalate of Iron (see No. 37) 20 Gm.

Heat iron strongly in empty sand bath until no further change is apparent upon stirring, and a sample is uniformly reddish brown when cold. (Preserve sample.)

37.—FERRI OXALAS (Wet Process):

 Sulphate of Iron
 20 Gm.

 Oxalic Acid
 10 Gm.

 Water of Ammonia
 Q.S.

 Water
 Q.S.

Dissolve the acid in a mixture of 25 C.c. of ammonia and 150 C.c. of water. Add sufficient ammonia to render mixture but slightly acid to test paper, and filter. Dissolve the iron in 300 C.c. hot water, and filter. Mix the two solutions. Wash the precipitate by decantation, and afterward upon a filter with water, until the washings are tasteless. Dry product between bibulous paper. (See No. 36.)

OXIDATION.

38.—Hydrargyri Oxidum Rubrum:

| Mercury. | | | | | | | 18 Gm. |
|-------------|--|--|--|--|--|--|--------|
| Nitric Acid | | | | | | | 6 Gm. |
| Water | | | | | | | 8 Gm. |

Dissolve 9 Gm. of mercury with gentle heat in the acid, and water previously mixed, and evaporate to dryness. Place the dry mass in capsule with balance of mercury, and heat until red vapors cease to rise. (Preserve sample.)

FUSION.

39.—Sulphuris Iodidum, U. S. P.:

| Washed | St | alp |)hı | ur | (N | To. | 4 | 2) | | | | 2 | Gm. |
|--------|----|-----|-----|----|----|-----|---|----|--|--|--|---|-----|
| Iodine | | | | | | | | | | | | | Gm. |

Mix well in mortar. Place in small flask, cork very



loosely, and apply gentle heat to darken mass, but not melt it. When color is uniformly dark, increase heat to produce liquefaction, and incline flask in different directions to recover any iodine that may have condensed on the inner side. When fusion has taken place, pour the mass quickly upon glass plate and let cool. (Preserve sample.)

CRYSTALLIZATION.

40. -- Sodii Pyrophosphas:

Sodium Phosphate 50 Gm.

Heat in capsule on water bath until dry mass remains, then transfer to empty sand bath and apply strong heat until a sample dissolved in water forms a perfectly white precipitate with test solution of silver nitrate. When all portions will thus act, remove heat, let cool, powder, and dissolve in 100 C.c. boiling water. Filter the solution when hot, and set aside twenty-four hours to crystallize. Drain and dry crystals. Evaporate mother liquor to obtain second-crop crystals. Drain, dry and add to previous lot. Preserve for making Ferri Pyrophosphas.

41.—Ferri et Ammonii Sulphas:

Solution Tersulphate of Iron (No. 48). 60 C.c. Sulphate of Ammonium 8 Gm. Diluted Sulphuric Acid 5 C.c.

Heat the solution of tersulphate to boiling, add ammonium salt, and, when dissolved, add the acid. Set mixture aside in well-covered dish in cool place for day or two to crystallize. Remove crystals from mother liquor, wash hastily with *little cold* water, dry between bibulous paper, and at once place in dry, cool, well-corked bottle. Obtain more crystals from mother liquor by evaporation. (Preserve sample.)



WET PROCESSES.

| 42.—SULPHUR LOTUM, U. | 5. | Ρ. | : | | | |
|-----------------------|----|----|---|--|--|--------|
| Sublimed Sulphur. | | | | | | 48 Gm. |
| Water of Ammonia | | | | | | |
| Water | | | | | | OS |

Add the sulphur to 48 C.c. of water previously mixed with the ammonia, and digest, with shaking, until next lesson. Then add 48 C.c. water, transfer to muslin strainer, and wash until drainings produce no precipitate in test solution of barium chloride. Let drain, dry in atmosphere, and powder with spatula. (Preserve for use.)

43.—Liquor Sodæ Chloratæ, U. S. P.: Carbonate of Sodium 20 Gm.

Water Q.S.

Mix lime well with 80 C.c. water, in bottle of 250 C.c. capacity, and cork. Dissolve sodium in 80 C.c. boiling water, and at once pour into first mixture. Cork tightly, and when cold add 10 C.c. water. Strain mixture through muslin quickly, allow precipitate to subside, and decant clear solution. Bottle at once.

GRANULATION.

44.—GRANULATED CHLORATE OF POTASSIUM:

Chlorate of Potassium 30 Gm. Boiling Water 100 C.c.

Dissolve the potassium, and filter quickly, washing through any crystals that may form on filter with hot water.



Stir constantly with rod, until cold. Collect crystals on filter, drain, dry between bibulous paper. Evaporate mother liquor to 25 C.c., stir until cold, collect, drain and dry second-crop crystals. Weigh total product, and ascertain loss. (Preserve sample.)

45.—Granulated Chloride of Ammonium:

| Crude Ammonium (| Chl | or | ide | | | 30 Gm. |
|------------------|-----|----|-----|--|--|---------|
| Boiling Water | | | | | | 75 C.c. |
| Water of Ammonia | | | | | | 2 C.c. |

Dissolve the chloride with water in flask, add the ammonia, and, after bringing the solution to boiling point, filter while hot. Evaporate, in capsule on water bath, to dryness, stirring constantly with glass rod. (Preserve sample.)

SOLUTION OF METAL IN ACID.

46.—Hydrargyri Sulphas:

| Mercury. | | | | | | 12 Gm. |
|-----------|--|--|--|--|--|--------|
| Sulphuric | | | | | | 18 Gm. |

Heat mercury with acid in capsule, stirring constantly until metal disappears. Continue heat until a dry, white salt remains. (Preserve sample.)

This preparation *must* be made in the fume closet.

SOLUTION OF OXIDE IN ACID.

47.—PLUMBI NITRAS:

| Oxide | of Le | ad | | | | | | 18 | Gm. |
|--------|-------|----|--|--|--|--|--|-----|-----|
| Nitric | Acid | | | | | | | 20 | Gm. |
| Water | | | | | | | | 100 | Gm. |

Heat ingredients together in capsule on sand bath until lead oxide is dissolved, filter solution, evaporate to 35 C.c., and set aside to crystallize; evaporate mother liquor to pellicle to obtain second-crop crystals. Drain, and dry between paper. (Preserve sample.)



WET OXIDATION.

48.—LIQUOR FERRI TERSULPHATIS, U. S. P.:

| Sulphate of Iron | ì. | | ٠ | | | | | 160 | Gm. |
|------------------|----|--|---|-----|---|----|----|-----|-----|
| Sulphuric Acid | | | ٠ | | | | | 30 | Gm. |
| Nitric Acid | | | | | ٠ | | | | |
| Distilled Water | | | | . ā | ā | 0. | S. | 100 | Gm. |

Mix sulphuric acid with 22 Gm. (16 C.c.) nitric acid and 100 C.c. distilled water in capacious capsule. Heat to boiling point, and add, one-fourth at a time, the sulphate of iron, stirring after each addition until effervescence ceases: Add few drops nitric acid. If fumes arise, continue adding acid cautiously until they cease, then apply heat until the solution becomes reddish brown and free from nitrous odor. Filter and add enough distilled water to make the whole weigh 40c Gm. (300 C.c.) (Preserve for use.)

49. - LIQUOR FERRI CHLORIDI, U. S. P. :

| Iron Wire | | | | | | 15 Gm. |
|-------------------|--|--|----|---|-----|---------|
| Hydrochloric Acid | | | | | | 86 Gm. |
| Nitric Acid | | | | | | |
| Distilled Water | | | āā | C | S.S | 100 Gm. |

Put iron in large flask, pour upon it 48 C.c. hydrochloric acid previously diluted with 25 C.c. distilled water, and let the mixture stand until effervescence ceases, (one week,) then heat to boiling, filter through paper, and, having rinsed the flask and iron wire with 10 C.c. boiling distilled water, pour the washings through filter. To the filtered liquid add 23 C.c. hydrochloric acid, and pour the mixture slowly and gradually in a stream into 6 C.c. nitric acid contained in large capsule. After effervescence ceases, apply heat by means of sand bath until the liquid is free from nitrous odor. Then test a small portion with freshly-prepared solution of ferricyanide of potassium. Should this produce a blue color,



add a little more nitric acid and evaporate off the excess. Finally add remaining 4 C.c. hydrochloric acid and enough distilled water to make solution weigh 100 Gm. (71 C.c.) (Preserve for use.)

BOILING.

50.—Potassi Carbonas:

Bicarbonate of Potassium 50 Gm.

Dissolve in 100 C.c. water, heat to boiling, and, after effervescence entirely ceases, evaporate to dryness on water bath, stirring constantly. (Preserve sample.)

MISCELLANEOUS.

51.—TINCTURA FERRI CHLORIDI, U. S. P.:

Solution Chloride of Iron (No. 49.)
. (25 C.c.) 35 Gm.
Alcohol (79 C.c.) 65 Gm.

Mix solution with the alcohol, and let stand in closely-covered vessel at least three months, then transfer to glass-stoppered bottles.

52.—Syrupus Ferri Iodidi, U. S. P.:

| Iron, in t | fine | wir | е. | | | | | | 2.5 | Gm. |
|------------|------|------|----|--|--|-----|----|----|-----|-----|
| Iodine . | | | | | | | | | 8 | Gm. |
| Sugar | | | | | | | | | 60 | Gm. |
| Distilled | W | rter | | | | . (| Э. | S. | 001 | Gm. |

Introduce the iron into small flask, add to it 20 C.c. distilled water and afterward the iodine; shake mixture occasionally, and let it stand until next lesson. Place sugar in capsule, and filter the solution iodide of iron into it. Rinse flask and iron with 9 C.c. distilled water, and pass washings



through filter into sugar. Stir the mixture with glass rod, heat to boiling point on sand bath, strain, and add enough distilled water to make the product weigh 100 Gm. (75 C.c.)

53.—LIQUOR FERRI CITRATIS, U. S. P.:

Solution Tersulphate of Iron (No. 48)

To the ammonia previously diluted with 100 C.c. cold water, add, constantly stirring, the solution of iron previously diluted with 500 C.c. cold water. Pour on wet strainer, allow precipitate to drain, then return it to the vessel and mix intimately with 600 C.c. cold water. Again drain on strainer, and repeat until washings cause but slight cloudiness with test solution chloride of barium. Let drain well. Transfer moist precipitate to capsule, add citric acid, and heat mixture on water bath to 60° C., stirring constantly until the precipitate is dissolved. Lastly, filter the liquid and evaporate at the above-mentioned temperature until it weighs 50 Gm. (40 C.c.) (Preserve for use.)

54.— Assay of Liquor Ferri Chloridi:

Solution Chloride of Iron (No. 49)

(15 C.c.) 20 Gm. Water of Ammonia Q. S.

Precipitate the solution of iron with an excess of ammonia, wash on filter with water, dry with gentle heat, ignite in small weighed capsule on sand bath, and ascertain weight. Record weight obtained. What should be the amount?

55.—OLEATUM ZINCI:

| Acetate of Zinc. | | | | | | 3 Gm. |
|------------------|--|--|--|--|-----|---------|
| Powdered Soap. | | | | | . / | 7.5 Gm. |
| Water | | | | | | |



Dissolve zinc in 250 C.c. cold water, and the soap in 125 C.c. hot water. When cold, add the soap solution to the solution of zinc, stirring briskly; drain the precipitate on muslin and wash several times with cold water. Dry on paper, well protected from dust and without heat. (Preserve sample.)

56.—ACID BORICUM:

| Borax | | | | | 10 Gm. |
|-------------------|--|--|--|--|--------|
| Hydrochloric Acid | | | | | 6 Gm. |
| Boiling Water | | | | | 24 Gm. |

Dissolve borax in boiling water, and filter while hot, mix with acid, and set the liquid aside for twenty-four hours in a cool place. Collect crystals, and wash with a *little* cold water. Then re-dissolve in five times their weight of boiling, distilled water. After day or two, collect crystals and wash with little water as before; dry in moderately warm place. (Preserve sample.)

SCALING.

57.—FERRI CITRATIS, U. S. P. :

Solution Citrate of Iron (No. 53) . . . 50 Gm.

Evaporate solution at temperature not to exceed 60° C. (140° F.) to the consistence of syrup, and spread thinly on plates of glass, so that when dry the salt may be obtained in scales. (Preserve for sample and use.)

58.—Ferri Pyrophosphas, U.S. P.:

| Citrate of Iron (No. 57) | | | 9 Gm. |
|------------------------------|-----|--|--------|
| Pyrophosphate of Sodium (No. | 40) | | 10 Gm. |
| Distilled Water | | | |

Dissolve the citrate in *distilled* water on water bath. To solution add the pyrophosphate and stir constantly until dis-



solved. Evaporate the solution on water bath, at temperature not to exceed 140° F. to consistence of syrup, and spread thinly on glass that product may be obtained in scales. (Preserve sample.)

DOUBLE DECOMPOSITION.

59.—Hydrargyri Iodidum Rubrum:

Corrosive Chloride of Mercury . . . 12 Gm. Iodide of Potassium 15 Gm. Distilled Water Q.S.

Dissolve the mercury in 200 C.c. hot distilled water, and the potassium in 40 C.c. cold distilled water; filter the solutions separately. Add the solution of mercury, when cold, to the solution of potassium, stirring constantly. Set aside until the precipitate has subsided, decant the mother liquor, wash the precipitate with 300 C.c. cold water, stirring it up well, let the precipitate subside and decant three times, collect the precipitate on a filter and wash with distilled water until the washings cease to give a precipitate with test solution of nitrate of silver, then dry on paper at a temperature not exceeding 40° C. (104° F). (Preserve sample.)

60. —FERRI FERROCYANIDUM:

Solution of Chloride of Iron (No. 49) 11.50 C.c. Ferrocyanide of Potassium 7 Gm. Distilled Water Q.S.

Dilute the solution of iron with 225 C.c. of water. Dissolve the potassium salt in 150 C.c. water; filter the solutions separately. Add the potash solution to the solution ferric chloride slowly, stirring constantly. Let the precipitate settle and wash several times by decantation. Transfer the magma to a well wetted strainer, or filter, and wash until washings are tasteless, let drain and dry at ordinary temperature, and reduce to fine powder. (Preserve sample.)











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